

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Patent Application of:  
Jens Ehlers et al.

Application No.: 09/787875

Confirmation No.: 8024

Filed: July 19, 2001

Art Unit: 1713

For: PREPARING A POLYMER

Examiner: R. Rabago

MS Amendment  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Dear Sir:

**DECLARATION**

I, Tim Dickner, Dr. rer. Nat., am a citizen of the Federal Republic of Germany and reside at Frankfurt, Federal Republic of Germany, hereby declare and say as follows:

1. I am a fully trained chemist, having studied chemistry at the universities of Münster and Braunschweig, Germany, and have received the following academic degrees:

Diploma in 1997,

Ph. D. in 2000

I am well acquainted with technical English.

2. From May 2001 I have been employed by Celanese AG as a head of a research laboratory. I worked exclusively in the field of catalyst research for polymerization of olefins from May 2001 to the present. Since 2004 I have been working in research projects for Ticona GmbH and since 2005 I have been employed by Ticona GmbH as well.

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3. In the polymer field, I am inventor up to now of at least 8 U.S. patents and patent applications and author of more than 20 publications and lectures.
4. In view of my qualifications as outlined above, I consider myself to be an expert and to be skilled in the polymerization art.
5. I am familiar with application Serial No. 09/787,875 and read US patent Nos. 5,633,419 and 4,910,272.
6. I had the following experiments conducted under my supervision. All the reactions were performed under argon with Schlenk-line technique and solvents were dried prior to use.
7. 

Example 1: Formation of unsupported catalyst:

Into a dried flask under argon with 170 ml of Exxsol 10 ml Isoprenylaluminum (20%) are added dropwise within 15 min at a steering speed of 200 rpm. Afterwards the temperature is lowered to 10°C. At a steering speed of 200 rpm 1.28 ml TiCl<sub>4</sub> in 2.56 ml Exxsol are added rapidly. The mixture is steered at 10°C for additional two hours and for 15 hours at room temperature subsequently. The catalyst is used for polymerization without further processing.
8. 

Example 2: Formation of supported catalyst:

In a dried flask under argon 10 g of silica are suspended with 170 ml of Exxsol. At a steering speed of 200 rpm, 10 ml Isoprenylaluminum (20%) are added dropwise within 15 min. The mixture is steered for additional 2 hours. Afterwards the temperature is lowered to 10°C. At a steering speed of 200 rpm 1.28 ml TiCl<sub>4</sub> in 2.56 ml Exxsol are added rapidly. The mixture is steered at 10°C for additional two hours and for 15 hours at room temperature subsequently. The catalyst is used for polymerization without further processing.
8. The Silica types used for the support were as follows: A) Grace 948 ( $d_{50} = 55 \mu\text{m}$ ); B) I NEOS CS5027 ( $d_{50} = 25 \mu\text{m}$ )

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9. **Polymerization:**

In a 2 l -steel-autoclave 1.5 l of Exxsol are placed. At a steering speed of 100 rpm the appropriate amount of catalyst (see table 1) together with 3.4 ml Isoprenylaluminum (20%) is added into the reactor. The reactor is brought onto reaction temperature (80°C) and at a steering speed of 250 rpm a constant ethylene pressure of 4 bar is applied. The polymerization is conducted up to the capacity of the reactor and the polymerization is stopped by venting of the ethylene pressure. The polymer is filtrated off and it is dried in vacuo at 80°C. The following results in Table 1 were obtained:

10. Table 1:

catalyst	1	2A	2B
Silica - d50	no - /	Grace 948 – 55µm	CS5027 – 25µm
catalyst amount	0.05 mmol	0.6 mmol	0.6 mmol
polymerization time	90 min	102 min	75 min
yield	236 g	297 g	304 g
activity [kg PE h <sup>-1</sup> mol <sup>-1</sup> ]	3150	290	405
d <sub>50</sub>	78 µm	305 µm	189 µm
bulk density	220 g/l	400 g/l	370 g/l

11. **Summary:**

The claimed catalyst system was supported on two different types of granular silica grades with a medium particle diameter of 55 and 25 microns respectively. In subsequent polymerization tests the supported catalyst 2A and 2B lead to totally different polymers compared to the original catalyst 1. The supported catalysts lead to significantly higher bulk densities and bigger particles.

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12. This is in contrast to examples in U.S. Patent No. 5,633,419 where granular supported catalysts yielded lower bulk density polymers (table 2). It is also in contrast to examples of U. S. Patent No. 4,910,272 where supported catalysts yielded lower bulk density polymers than unsupported catalysts (table 3).

12. Table 2

example	comparative 11	comparative 10
silica - d <sub>50</sub>	45G - 45 $\mu$ m	30G - 30 $\mu$ m
polymer d <sub>50</sub>	111 $\mu$ m	114 $\mu$ m
Bulk density	237 g/l	245 g/l

13. Table 3

example	comparative A	Example 1	Example 6
silica - d <sub>50</sub>	No silica	16 $\mu$ m	range 75-106 $\mu$ m
polymer d <sub>50</sub>	292 $\mu$ m	166 $\mu$ m	454 $\mu$ m
Bulk density	282 g/l	149 g/l	112 g/l

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States

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Code and that such wilful false statements may jeopardize the validity of the above-identified application or any patent issues thereon.

  
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Tim Dickner

24.10.2005  
Date: